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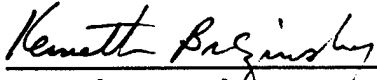
Air Force Systems Command

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FUELS COMBUSTION RESEARCH

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ABSTRACT

Extensive progress has been made in all aspects of the program during the subject period. The efforts in understanding the oxidation of aromatic components of JP fuels led to the completion of a study of the oxidation of 1-methylnaphthalene, a species known to contribute extensively to soot formation. This study is the first to report detailed oxidation results for any polycyclic aromatic hydrocarbon despite the presence of these compounds in many practical fuels. Based on the observed intermediate species in these flow reactor experiments a mechanism has been proposed and published. This mechanism is strongly analogous to one published on toluene oxidation.

That aspect of the work directed towards the understanding of aliphatic fuel oxidation and pyrolysis concentrated on methylcyclohexane (MCH), an endothermic fuel for high speed flight. Its combustion properties and that of a blend with toluene were analyzed. Experimental results revealed the major intermediate species during both the oxidation and pyrolysis of MCH were ethene, butadiene, methane and propene. Since these intermediates were also studied in the program, a rather complete understanding of MCH processes has been obtained. MCH/toluene blends show that MCH accelerates the toluene fuel decay, but not vice-versa, and that neither fuel's mechanism is affected by the other.

Although the efforts on soot formation has diminished in favor of those related to fuel fouling under sub- and super-critical conditions, some contributions had been made in further understanding of the fuel smoke height test. Experiments have led to the conclusion that a critical temperature for soot particle nucleation exists irrespective of the fuel species for combustion conditions. This concept explains how the smoke height occurs and how flame temperature affects the smoke height. The analysis of the smoke height proposed could have important ramifications in understanding soot formation in many other systems.

Degradation studies of toluene, methylcyclohexane and toluene/methylcyclohexane mixtures were conducted at unique combinations of temperatures and pressures in a sub/super-critical liquid flow reactor made of nickel tubing. It has been found that nickel particles in the presence of supercritical methylcyclohexane facilitate the growth of graphitic filaments that might otherwise form only at much higher temperatures. The observations suggest that graphitic filaments may contribute to fuel line clogging when the fuel lines are made of nickel containing stainless steel. Considering the fundamental understanding obtained, further examination of the contribution of fuel line material and the contribution of homogeneous fluid phase hydrocarbon cracking to deposit formation are the continuing efforts in this part of the integrated research program for gas turbine fuels, particularly those to be used in the next generation engines.

I. Introduction

This report deals with the objectives of the center of excellence in fuels research developed at Princeton under the AFOSR Grant. This section will not give an extensive review and history of the efforts previous to this most recent initiative, nor dwell on the more than one hundred publications which have been a result of continued AFOSR support. Many of these publications have been identified in the previous AFOSR reports and presentations. Rather, in the following sections the principal tasks of the program are discussed more succinctly:

1. aromatic pyrolysis and oxidation
2. aliphatic hydrocarbon pyrolysis and oxidation kinetics, now particularly directed towards endothermic fuels.
3. soot formation and destruction mechanisms
4. fuel degradation and deposit formation under sub- and super-critical operating conditions.

II. Research Progress

A. The Oxidation and Pyrolysis of Aromatic Hydrocarbons

The early part of this program was devoted to investigations of the oxidation characteristics of aromatics and considered benzene, mono-alkylated benzenes particularly toluene and the di-alkylated benzenes, particularly o, m and p-xylenes. This research led to numerous publication (1-12). The major focus of the research under the subject grant period was on the oxidation of 1-methylnaphthalene and has provided the first detailed oxidation results for any polycyclic aromatic hydrocarbon (PAH), despite the presence of PAH's in many practical fuels. The results of this study can be summarized as follows.

Detailed intermediate species profiles for the oxidation of 1-methylnaphthalene were obtained by gas-phase sampling from the atmospheric-pressure flow reactor operating at about 1170 K. No evidence was found to indicate that fuel consumption through ring reactions was competitive with consumption through attack of the methyl side chain. Based on the observed intermediate species profiles, a mechanism was proposed for the oxidation of 1-methylnaphthalene which was strongly analogous to a previously developed mechanism for the oxidation of toluene under flow reactor conditions. In this mechanism, the resonantly stabilized 1-naphthylmethyl radical undergoes radical-radical reactions to form 1-naphthaldehyde, which decomposes to 1-naphthyl radical. Naphthalene and 1-naphthyl radical are oxidized to 1-naphthoxy radical, which decomposes to indenyl radical. Further reaction and decomposition results in the formation of phenylacetylene, styrene, phenyl radical, and acetylene. This mechanism is consistent with the observed sequential formation of the major aromatic intermediates 1-naphthaldehyde, naphthalene, indene, phenylacetylene, and benzene. Other pathways to the formation of minor intermediates such as 2-methylnaphthalene, acenaphthylene, 2-naphthol, and 1-methylindene are considered in this mechanism and the detailed results are reported in Refs. 13 and 14.

B. The Oxidation And Pyrolysis Of Aliphatic Hydrocarbons

Since endothermic fuels have become of interest to the Air Force, this aspect of the program took a change in direction to consider the pyrolysis and oxidation characteristics of these types of fuels.

It is known that methylcyclohexane (MCH), the most commonly thought of endothermic jet fuel, decomposes to hydrogen and toluene in presence of a catalyst. In order to understand the impact of this cyclic aliphatic compound on the combustion characteristics of aromatic fuels in jet propulsion engines, pyrolysis and oxidation studies of pure MCH and, oxidation studies of MCH/toluene blends were conducted over the temperature range of 1050-1200K in the turbulent flow reactor.

Although some results of this study in retrospect may seem somewhat obvious, particularly in regard to the blends, they are enlightening and encouraging in that blend studies may follow a pattern so that detailed studies of variations of the blend components may be unnecessary. Examination of pure MCH during pyrolysis and oxidation revealed that ethane, butadiene, methane and propene were the major stable intermediates. Since the pyrolysis and oxidation characteristics of these intermediates have already been studied in an earlier AFOSR program, the overall mechanistic character of these processes now appear to be understood.

The oxidation experiments of MCH/toluene blends have led to three significant findings relevant to component selection in multi-component JP fuels. They are:

- 1) Toluene, commonly thought to be a reaction inhibitor, has little effect on the rate of oxidation of MCH.

- 2) Early in the oxidation process MCH produces an extensive radical pool which is not only sufficient to insure MCH's rapid oxidation, but also to serve to accelerate the oxidative pyrolysis of toluene.

- 3) When oxidized as a blend, MCH/toluene, despite sharing a radical pool, each appear to follow their own oxidation mechanism as pure species.

C. Soot Formation And Destruction Process

A major aspect of the soot problem that was resolved during the subject grant period was with regard to the critical temperature for soot nucleation. By measuring the temperature along the center-line of a burning fuel jet which had been diluted with an inert gas until it was no longer luminescent, it was possible to establish a critical temperature at which a given fuel would no longer soot (15). The range of temperatures observed for the fuels evaluated in this manner was extremely narrow. The preliminary results reported in the earlier report were indeed validated by more extensive experimentation. These results showed about 100 K variation in the critical temperature to prevent sooting and the order would indicate that the dilution technique could have affected the results to give this variation. The fuels that had the least tendency to soot demanded the least amount of dilution and gave the highest critical temperature. It would appear then that a single critical temperature for soot nucleation in diffusion flame tests exists and that the actual temperature is in the 1600-1700 K range regardless of the fuel. Since the mechanism for the formation of the initial fuel particle is the same regardless of the fuel, some high activation process in the soot formation mechanism would appear to control initial particle nucleation.

The first complete explanation of the temperature in sooting diffusion flames, particularly those using circular fuel jets, was developed during the contract period. Essentially the critical temperature isotherm, say 1650 K, is the point at which soot particles nucleate. The soot volume fraction grows by mass addition to the particles until the particles enter the higher temperature flame front. As the volumetric flow rate is increased, the separation distance of the flame and critical temperature isotherms remains essentially the same, but the amount of particles nucleated increases the soot volume fraction so that the particles penetrate the flame front and a smoke height is reached. Different fuels will form different numbers of nucleated particles at the 1650 isotherm, so different smoke heights are obtained for different fuels. When the fuel is diluted with an inert gas, the flame temperature drops, and the 1650 isotherm moves closer to the flame temperature isotherm.

Thus the growth period of the nucleated particles decreases, and the soot volume fraction entering the flame is reduced. To create a smoke point for this diluted condition, the volumetric flow rate of the fuel-inert mixture must be increased so that more particles will form.

What is important about the above analysis of sooting gaseous fuel jets is that the concepts proposed could indeed have broad application.

D. Supercritical Fluid And High Temperature Liquid Fuel Degradation

An apparatus was developed under the program for the study of solid deposit formation in the fuel transfer lines of aircraft jet engines. A study of toluene, methyl cyclohexane, and toluene/methyl cyclohexane mixtures at unique combinations of temperatures and pressures was begun during the subject period.

A hydrocarbon fluid of interest is driven through a heated reaction tube by high pressure piston pumps capable of delivering the fluid at pressures up to 5000 psi. The heated reaction tube is, but is not limited to, nickel capillary tubing which has a nominal inner diameter of 0.43mm. This size tubing enables the fluid to be heated and cooled rapidly, and still maintain adequate residence times in the heated zone by choosing the heated coil's length and pump flow rate. Analysis has shown that the various parameters chosen have created the desired idealized plug flow reactor in which each parcel of fluid experiences identical flow histories. The heated reaction zone is kept at a constant temperature with a Techne fluidized sand bath. The bath's electric heaters can subject the heated coil to temperatures up to 600C.

The temperature is regulated in the fluidized bed by an electronic temperature controller. Thermocouples monitor the temperature of the heating elements and at the center of the fluidized bed and the fluid temperature at the entrances and exits of the heating and cooling coils. The pressure is monitored upstream of the heated coil with a standard pressure gauge and upstream and downstream of the heated coil with a mV output pressure

transducer. Pressure is regulated with needle valves in combination with a spring loaded back pressure device.

The apparatus described above was used to examine changes in the chemical composition of toluene, methyl cyclohexane and mixtures of these two species in order to deduce the chemistry responsible for fuel line deposit formation. Figure 1 graphically portrays the matrix of experimental conditions examined. The figure contains lines indicating the critical temperature of toluene and methyl cyclohexane, as well as lines indicating the critical pressures. To date, most of the experiments conducted have been at temperatures and pressures above the critical points of the pure fluids and probably above those for the toluene/methyl cyclohexane mixture. Figure 2 indicates that residence times have been varied from almost 20 minutes to as little as almost 1 minute.

As can be observed in Figure 1, one set of systematic experiments examining the chemical changes in pure methyl cyclohexane has been conducted as a function of temperature. This set is shown on the figure by solid black squares. As the methyl cyclohexane is heated it approaches temperatures at which solid material forms and clogs the reactor tube. Cases where this clogging has been observed are shown on the figure and will be discussed later in this report.

The experiments conducted at temperatures just preceding that which causes clogging have yielded fluid samples that were analyzed by gas chromatography - Fourier Transform spectroscopy. The preliminary results indicated that the methyl cyclohexane fragments on heating to produce light gases such as methane and a variety of four- and five-carbon species. In order to determine their identities, these latter species are being examined more closely during the new grant period awarded. Also, benzene appears to have formed. What did not appear to be formed are large molecular species such as polynuclear aromatics or polymers that would constitute the precursor species to deposit formation. Investigation of the liquid phase chemistry now continues in the newly awarded grant.

Two of the three experiments that led to clogging yielded solid samples suitable for analysis. The first experiment, indicated on the figure by a solid black up triangle marked "clogged", was conducted with a 90% toluene, 10% methylcyclohexane mixture at 797K, 4.63MPa and a residence time of 1.05 minutes. The second, indicated on the figure by a solid black circle labeled "clogged", is the methyl cyclohexane experiment at 657K, 4.14 MPa and residence time of 4.12 min.

These two experiments exhibited the onset of particulate formation clogging by a rapid rise in the fluid pressure as the blockage developed in the nickel tubing. As this pressure rise occurred, the flow rate was drastically increased in order to drive the blocking material out of the tubing. The black powdery material that exited the reactor in the reactant fluid was captured and subsequently analyzed using transmission electron microscopy (TEM) and energy loss and electron diffraction spectroscopy.

Analysis was conducted with a CM 20 Philips TEM. The TEM and energy loss analysis indicated that the black powdery material consisted of filaments of graphitic platelets aligned parallel to the long axis of the filament. The EDAX (electron diffraction) analysis also indicated that the filaments contained, occluded within the filament, nickel particles. Copper was also shown to be present but only in the supporting grid necessary for the TEM analysis. Photographs were subsequently generated from the TEM analysis.

In Figure 3 a typical filament about 10000nm (10 microns) long and approximately 100 nm in diameter can be seen. Occluded within the filament is a diamond shaped particle subsequently shown to be nickel crystal. A magnified image of the particle and the surrounding material is shown in Figure 4. The surrounding material exhibits light and dark planar regions with an interplanar spacing characteristic of graphitic layers. The graphitic layers parallel the surface morphology of the nickel particle.

Another view of the graphitic filaments is shown in Figure 5. Again the long filaments contain diamond shaped particles and may even have such particles anchored at

the end of the filament. An electron diffraction pattern obtained from one of the particles contained in the middle of the filament identifies the particle as nickel.

Three unusual characteristics exhibited by these filaments appear to be unique to the ones shown in the photos. First, the whole length of the filament is composed of a gently curving rod without any kinks or sharp changes in direction. Kinks generally are sites of weakness in a fiber and lead to fracture at the kink location. Secondly, the nickel particles are contained within the length of the filament not just at the ends. The literature on this subject indicates that metal particles such as nickel occur at the end of the filament and serve as the base from which the filament grows. Graphitic filaments as produced generally have not been observed to contain nickel particles within, especially particles that appear to be faceted. Thirdly, although there appears to be the hint of a hollow core in these filaments, they are primarily solid rodlike structures. Graphitic filaments generally contain a substantial hollow center core. A unique characteristic of these filaments not obvious from the photos is that the conditions of production involve higher pressures and much lower temperatures, i.e., nominally 800K rather than 2000K, than have been required in other processes.

Subsequent to the generation of the above mentioned photos, the authors met with Dr. Yao of the Princeton Materials Institute to discuss the unique quality of the filaments. Dr. Yao pointed out that filaments described in the literature were very different than those produced. Based on the discussion and a subsequent literature examination, a presentation was formulated and made at the AFOSR Supercritical Fuels/Combustion Workshop at Wright Laboratory. The above mentioned photos of the filaments were presented as were the fabrication conditions. At that meeting the results were communicated to Dr. F. L. Abrams of the Structural Materials Branch of Wright Laboratory. Copies of the photographs were given her. The copies were relayed to Dr. J. Hager of the same Branch. A subsequent telephone conversation with Dr. Hager ensued within the following month. The importance of these fibers for structural reinforcement was discussed. Also discussed was the importance of

graphitic filaments to the Air Force and the relationship between Wright Laboratory and a small company, name unknown to us, that would commercialize graphite filament production techniques developed at Wright Laboratory. As a result of the discussion with Dr. Hager, it was decided that a patent application was timely and would protect the discovery and the interests of the Air Force. Although written, Princeton University has not submitted the patent application.

The essential discovery of importance to the Air Force is a method of fabrication of graphitic filaments in the presence of metal particles when exposed to hydrocarbon fluids either in the supercritical state, i.e. at high pressures and at temperatures above the critical point pressures and temperatures and, possibly when exposed to fluids at high pressure, and modest temperatures so that the hydrocarbons remain in the liquid state. It has not been the objective of this effort to develop a method of producing carbon fibers for practical use. The research undertaken surprisingly led to conditions under which unique carbon fibers could be grown. Since the main thrust of the research has been, and still is, aimed at understanding precursor formation to deposits from gas turbine fuels, the investigation of the formation of graphitic filaments is not being pursued within this program.

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IV. AREAS OF LIAISON WITH THE AIR FORCE

A special interaction with the research efforts at Wright-Patterson AFB under the direction of W.M. Roquemore was developed. Dr. Roquemore's group focuses on complementary aspects of understanding the fuel fouling tendency of fuels. Coordination

of Princeton experiments with Drs. Roquemore and Edwards at Wright-Patterson will permit the development of a "systems" view of advanced aircraft engine fuel fouling. Efforts at establishing a coordinated research effort have already begun and (during the subject grant) consisted of numerous telephone conversations, the exchange of proposals and pre-proposals, and visits to the Air Force Base by Dr. Brezinsky to discuss reactor design with both Air Force and University of Dayton personnel. Subsequently there was participation of the Princeton group in the Workshop on Supercritical Phenomena at WPAFB. This participation led to conversations with the Materials group at WPAFB who showed interest in the carbon fibers formed in the Princeton supercritical flow reactor. Contacts have been made with material scientists at the AF Phillips' Laboratory.

With respect to soot phenomena and oxidation of aromatics three suppliers of jet fuels, Texaco, BP and Shell, showed great interest in the Princeton results. Liaison has been maintained with other AFOSR contract monitors, in particular, UTRC, Penn State and Georgia Tech.

V. PUBLICATIONS

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"Effect of Oxygen Addition to a Near Sooting Ethene Inverse Diffusion Flame," Comb. Sci. and Tech. 81, 207 (1992).

"Flame Temperature, Fuel Structure and Fuel Concentration Effects on Soot Formation in Inverse Diffusion Flames", Combust. and Flame 90, 269 (1992).

"Pyrolysis and Oxidation Studies of Methylcyclohexane," Eastern and Central States Section/The Combustion Institute Meeting, Paper No. 83, March 1993.

"The High Temperature Oxidation of Naphthalene: A Benzene Analog?," Eastern and Central States Section/The Combustion Institute Meeting, Paper No. 88 March 1993.

"Oxidation Studies of Methylcyclohexane/Toluene Blends", Eastern States Section/The Combustion Institute Meeting, Oct. 25, 1993, Paper No. 16.

"A New Method of Producing Graphitic Filaments", Disclosure of New Technology, Princeton University, April 12, 1994.

"An Experimental Study of the High Temperature Oxidation of 1-Methylnaphthalene", Christopher R. Shaddix, Ph.D. Thesis, Princeton University, Mechanical & Aerospace Engineering Department, June 1993.

"An Experimental Study of Supercritical Methylcyclohexane Pyrolysis", Gary T. Davis, M.S.E. Thesis, Princeton University, Mechanical & Aerospace Engineering Department, November 1994.

"Flow Reactor Studies of the Pyrolysis and Oxidation of Methylcyclohexane", Stephen P. Zeppieri, M.S.E. Thesis, Princeton University, Mechanical & Aerospace Engineering Department, June 1994.

VI. PRESENTATIONS

"Benzene/Toluene Oxidation Models for Combustion Applications" Special Combustion Seminar, University of Southern California, Los Angeles, CA, December 13, 1991.

"Oxidation Studies of Aromatic Polycyclic Aromatic Hydrocarbons", Sandia Technical Seminar, Sandia Combustion Facility, Livermore, CA, December 17, 1991.

"Fuels Combustion Research", AFOSR Contractors Meeting, San Diego, California, June 19, 1992.

"High Pressure Flow Reactor Studies", University of Dayton, Dayton, Ohio, August 12, 1993.

"Reactions in Supercritical Fuels", Supercritical Fuels/Combustion Workshop, Wright Laboratory, Dayton, Ohio, February 7, 1994.

"Fuels Combustion Research", A.F.O.S.R. Contractors Meeting, Lake Tahoe, Nevada, June 9, 1994.

"Fuel Line Deposit Formation and Its Combustion Consequences", General Electric Research Center, Schenectady, N.Y., July 6, 1994.

"Fuel Line Deposit Formation and Its Combustion Consequences", Invited Presentation, Centre National de la Recherche Scientifique, Orleans, France, September 29, 1994.

VII. PERSONNEL

Irvin Glassman
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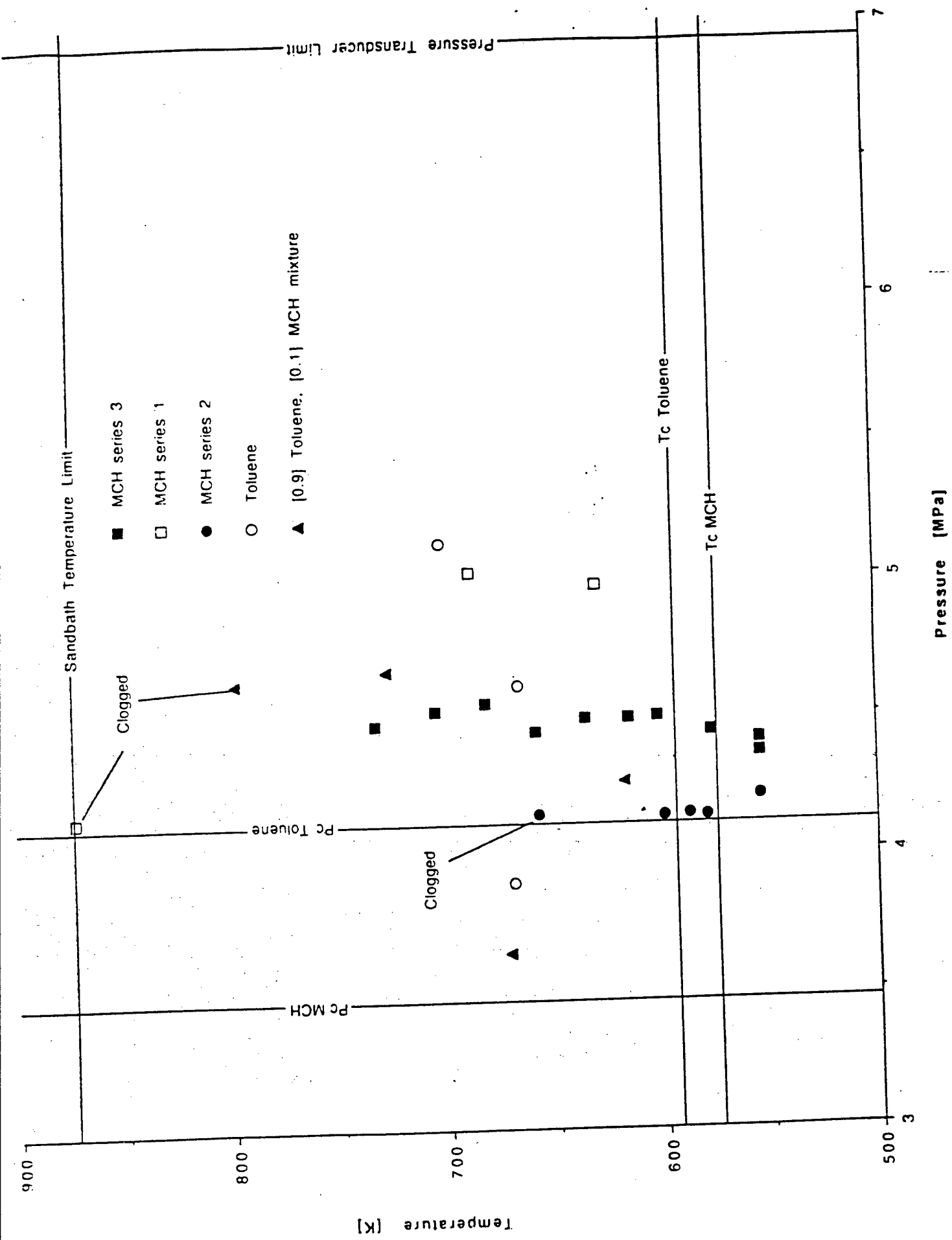


Figure 1

Residence Time vs. Temperature

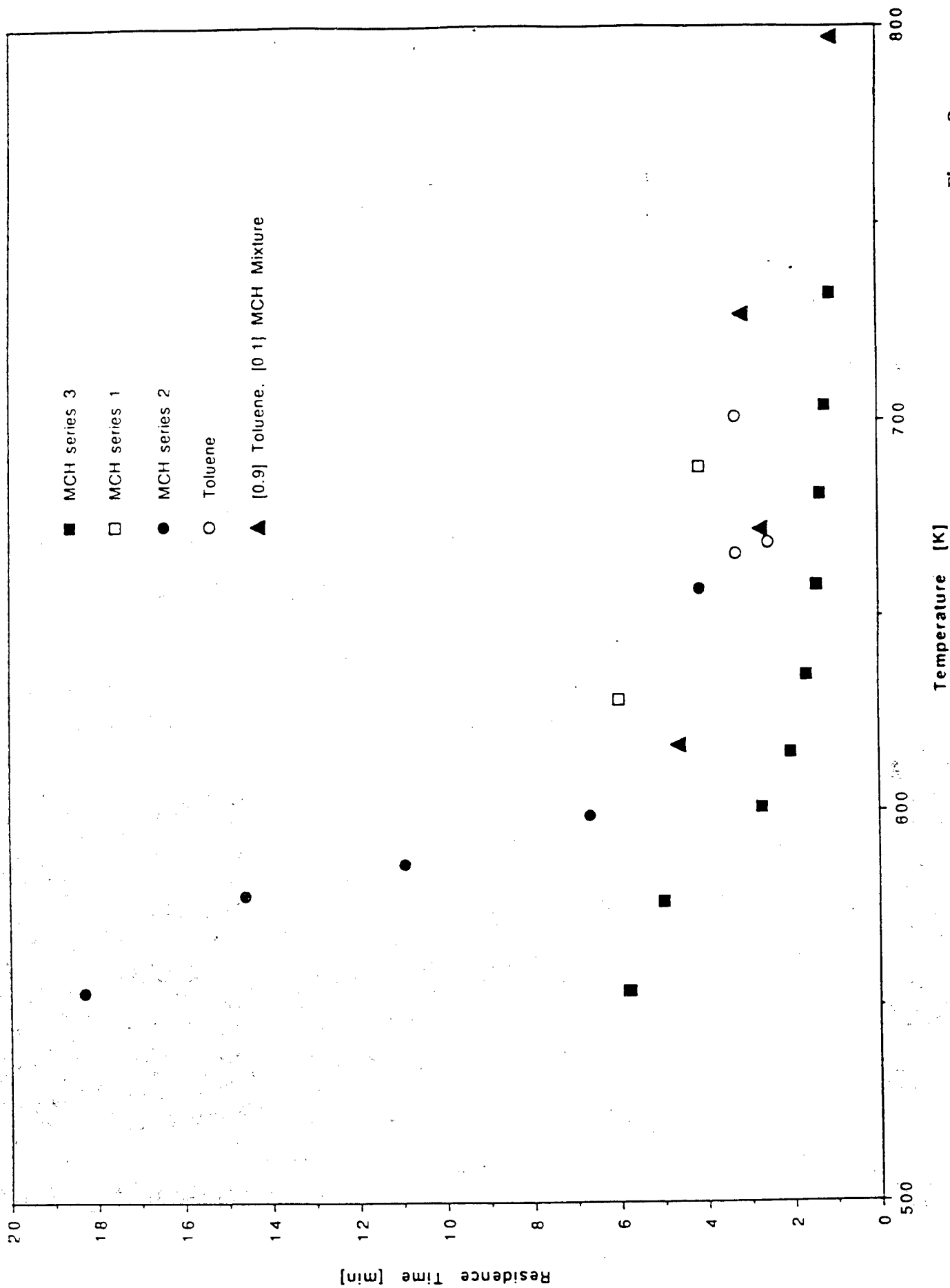


Figure 2

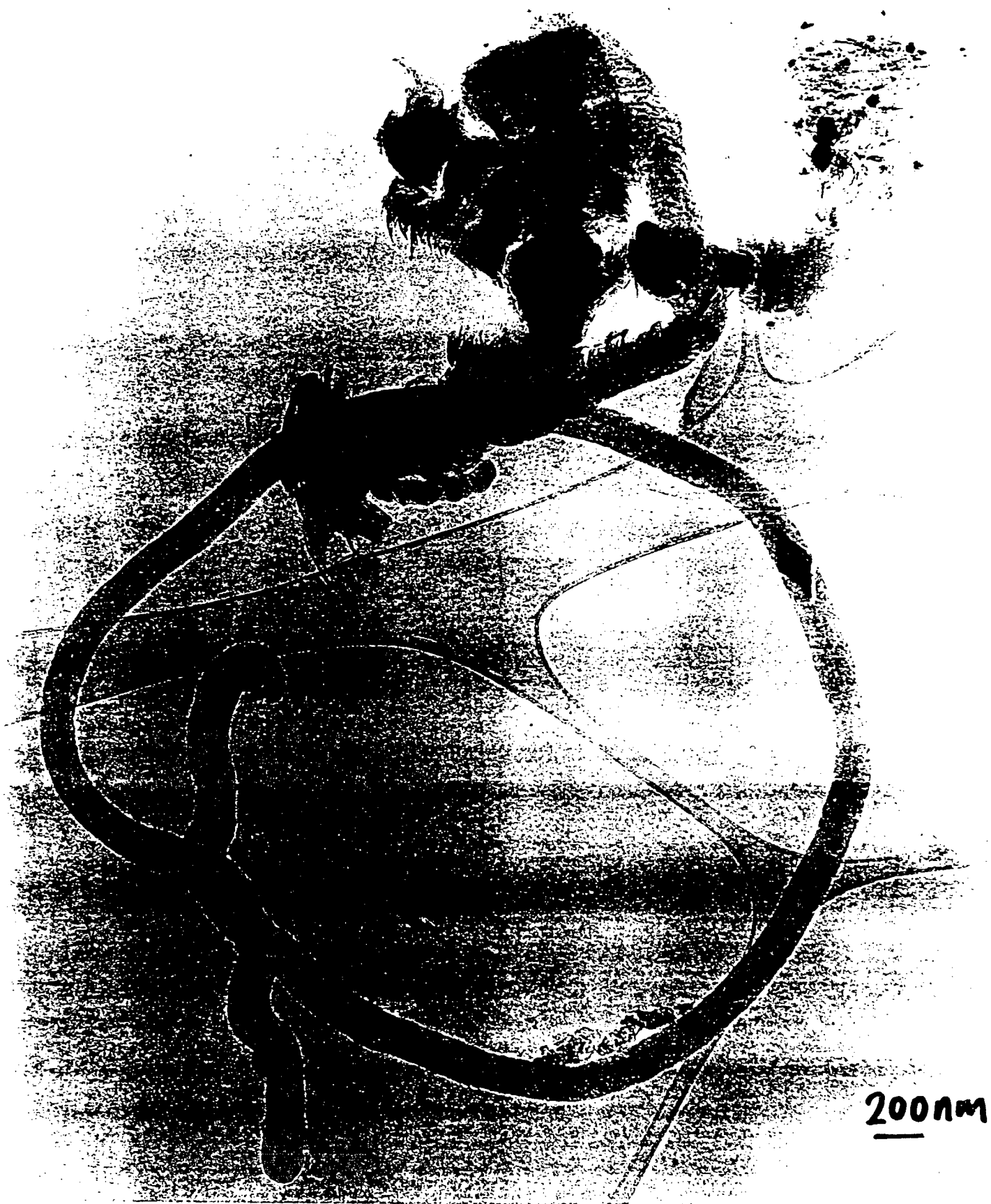


Figure 3

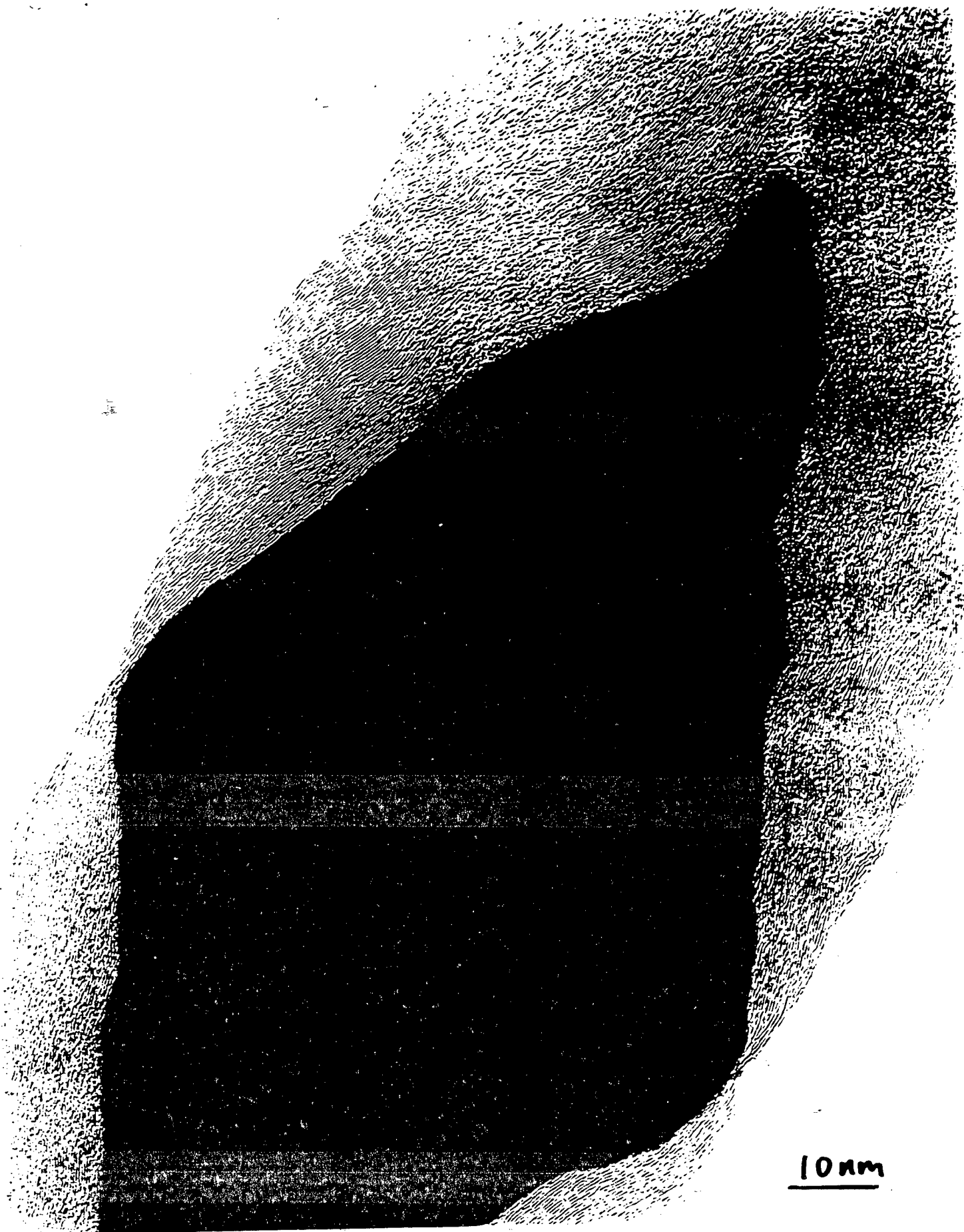


Figure 4

220K6788



200nm

Figure 5